

जल एवं अपशिष्ट जल के नमूने लेना और
परीक्षण (भौतिक एवं रसायनिक) की पद्धतियाँ

भाग 79 सिल्वर

**Methods of Sampling and Test
(Physical and Chemical) for Water
and Wastewater**

Part 79 Silver

ICS 13.060.50

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भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS

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FOREWORD

This Indian Standard (Part 79) was adopted by the Bureau of Indian Standards, after the draft finalized by the Water Quality Sectional Committee had been approved by the Chemical Division Council.

On an average, the silver being present in the crust of the earth is 0.08 ppm, in soils it is less than 0.01 ppm to 0.5 ppm, in streams its around 0.3 $\mu\text{g/l}$, and in case of ground water it is less than 0.1 $\mu\text{g/l}$. Silver occurs naturally and in combination with the elements that are non-metallic such as horn silver (AgCl) and argentite (Ag_2S). The ores of copper and lead may also yield considerable amount of silver. Silver is widely used in batteries, jewellery, silverware, mirrors and photography. In the seeding of clouds, silver iodide has been used and to a limited extent, silver oxide is used as a disinfectant for water.

In acidic water, silver ion predominates and in case of water with high chloride, a series of complexes would be expected. Silver is non-essential in case of animals and also plants. Silver can cause argyria which is a permanent, blue gray discolouration of the skin and eyes which gives ghostly appearance. Concentrations being in the range of 0.4 mg/l to 1 mg/l have caused pathological changes in the liver, kidneys and rats spleen. Toxic effects with fish present in fresh water habitat have been observed at concentrations as low as 0.17 $\mu\text{g/l}$. In case of freshwater aquatic life, total amount of recoverable silver should not exceed 1.2 mg/l.

Under the general title ‘Method of Sampling and Test (Physical and Chemical) for Water and Wastewater’ IS 3025 has been published in several other parts. This part of IS 3025 covers four test methods for determination of silver in water and wastewater.

In preparation of this standard considerable assistance has been derived from method no. 3111B, C, and 3113 B of Standard Methods for Examination of Water and Wastewater 23rd Edition 2017 published by American Public Health Association.

The composition of the committee responsible for the formulation of this standard is listed in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

*Indian Standard***METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER****PART 79 SILVER****1 SCOPE**

This standard (Part 79) describes the following four methods for the determination of silver in water and wastewater:

- a) Direct air-acetylene flame method;
- b) Extraction/air-acetylene flame method;
- c) Electrothermal atomic absorption spectrometric method; and
- d) Inductively coupled plasma method.

2 REFERENCES

The following standards contain provisions which through reference in this text constitute provisions of this standard. At the time of publications, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
IS 3025 (Part 2) : 2019	Methods of sampling and test (Physical And Chemical) for water and wastewater: Part 2 determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP - OES)
IS 3025 (Part 65) : 2022	Methods of sampling and test (Physical and Chemical) for water and wastewater: Part 65 application of inductively coupled plasma mass spectrometry (ICP - MS) — Determination of 62 elements
IS 7022 (Part 1) : 1973	Glossary of terms relating to water, sewage and industrial effluents: Part 1
IS 7022 (Part 2) : 1979	Glossary of terms relating to water, sewage and industrial effluents: Part 2
IS 17614 (Part 1) : 2021	Water Quality — Sampling: Part 1 Guidance on the design of sampling programmes and sampling techniques
IS 17614 (Part 3) : 2021	Water Quality — Sampling: Part 3 Preservation and handling of water samples

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 7022 (Part 1) and IS 7022 (Part 2) shall apply.

4 SAMPLING AND PRESERVATION

Sampling and sample preservation shall be done as prescribed in IS 17614 (Part 1) and IS 17614 (Part 3).

5 DIRECT AIR-ACETYLENE FLAME METHOD**5.1 Principle**

The silver content of the sample is determined by atomic absorption spectrophotometry. For dissolved silver, the filtered sample is directly aspirated to the atomizer. For total recoverable silver, a pre-treatment with HCl is carried out prior to aspiration of the sample. This method is applicable in the range from 0.1 mg/l to 4 mg/l of silver. However, the concentration range will vary with the sensitivity of the instrument used.

5.2 Apparatus**5.2.1 Atomic Absorption Spectrometer and Associated Equipment**

5.2.2 Use burner head as recommended by the manufacturer.

5.2.3 Standard Volumetric Glassware**5.3 Reagents**

5.3.1 Unless otherwise specified, only AR grade chemicals should be used for all the tests.

5.3.2 Air, clean, dried and free from oil, water and other foreign substances. The source may be a compressor or commercially bottled gas.

5.3.3 Acetylene, standard commercial grade.

5.3.4 Silver-free distilled water should be used for preparing standards and reagent solution.

5.3.5 Hydrochloric Acid (HCl), 1 percent, 10 percent, 20 percent (all v/v), 1 + 5, 1 + 1, and concentrated.

5.3.6 Nitric Acid (HNO_3), 2 percent (all v/v), 1 + 5, 1 + 1, and concentrated.

5.3.7 Stock Silver Solution

Take 0.1575 g of silver nitrate in 100 ml water. Now add 10 ml of concentrated nitric acid and make up the volume to 1 000 ml (1 ml = 100 μg of Ag).

5.3.8 Standard Silver Solution

Prepare a series of standard silver solutions in the optimum concentration range by appropriate dilutions of stock silver solution (5.3.7) with distilled water (5.3.4) containing 1.5 ml of concentrated nitric acid.

5.4 Procedure

5.4.2 Instrument Operation

5.4.2.1 It is difficult to formulate instructions as applicable to every instrument, because of differences between makes and models of atomic absorptions spectrometers. See manufacturer's operating manual.

5.4.2.2 Install a hollow-cathode lamp for silver in the instrument and set the wavelength at 328.1 nm. Set the slit width as suggested by manufacturer for silver being measured. Turn on the instrument and apply current to hollow cathode lamp for 10 min to 20 min to stabilize the energy source. After adjusting the wavelength, install burner head (5.2.2). Turn on the air (5.3.2) and adjust the flow rate as suggested by the manufacturer to give maximum sensitivity for silver being measured. Turn on acetylene (5.3.3) and adjust flow rate. Ignite the flame and let it stabilize for a few min.

5.4.2.3 Aspirate a blank consisting of distilled water (5.4.4) containing 1.5 ml of concentrated nitric acid. Set zero the instrument. Aspirate the silver standard solution. Check the aspiration rate of nebulizer and adjust it to obtain maximum sensitivity. Obtain maximum response by adjusting burner position vertically and horizontally. Aspirate the blank and re-zero the instrument. Aspirate standard silver solution near middle of linear range. Prepare a fresh same standard and with a new hollow cathode lamp, record the absorbance of this standard. Check consistency of instrument setup and aging of hollow-cathode lamp and standard referring to these data on subsequent determinations of silver.

5.4.2.4 The instrument is now ready to operate. At the end of analysis, first switch off acetylene than air.

5.4.3 Standardization

Prepare at least three standard silver solutions containing lower concentration 0.1 mg/l Ag to

bracket the expected metal concentration of a sample. Prepare a reagent blank of 100 ml volume with distilled water (5.3.4) containing 1.5 ml of concentrated nitric acid (5.3.6). Aspirate the reagent blank and carry out zero adjustment. Aspirate blank and zero the instrument. Aspirate each standard solution in turn into the flame and record the absorbance readings. Most modern instruments are equipped with micro-processors and digital read-out which permit calibration in direct concentration terms.

5.4.4 Analysis of Sample

Rinse nebulizer by aspirating distilled water (5.4.4) containing 1.5 ml concentrated nitric acid per litre and zero instrument. Aspirate a sample and determine its absorbance.

5.5 Calculation

Prepare a calibration curve on linear graph paper by plotting absorbance reading versus their reading. Alternatively, read the concentration directly from the instrument read-out if the instrument is so equipped. If sample has been diluted, multiply by the appropriate dilution factor.

6 EXTRACTION/AIR-ACETYLENE FLAME METHOD

6.1 General

The given method is suitable for the detection of low concentrations of silver in water and wastewater. This method uses ammonium pyrrolidine dithiocarbamate (APDC) as the chelating agent, followed by extraction into methyl isobutyl ketone after the aspiration into an air-acetylene flame.

6.2 Apparatus

6.2.1 Atomic Absorption Spectrometer and Associated Equipment

6.2.2 Use burner head as recommended by the manufacturer.

6.3 Reagents

6.3.1 Unless otherwise specified, only AR grade chemicals should be used for all the tests.

6.3.2 Air, clean, dried and free from oil, water and other foreign substances. The source may be a compressor or commercially bottled gas.

6.3.3 Acetylene, standard commercial grade.

6.3.4 Silver-free distilled water should be used for preparing standards and reagent solution.

6.3.5 Methyl Isobutyl Ketone (MIBK), reagent grade.

6.3.6 Nitric Acid, concentrated, ultrapure.

6.3.7 Sodium Sulphate, anhydrous.

6.3.8 Ammonium Pyrrolidine Dithiocarbamate Solution

Dissolve 4 g of Ammonium pyrrolidine dithiocarbamate in 100 ml water. If necessary purify the salt with an equal volume of MIBK. In a separating funnel, shake it for 30 sec. Withdraw a lower portion and discard MIBK layer.

6.3.9 Water Saturated MIBK

In a separating funnel, mix one part of purified MIBK with 1 part of water. Shake it off for 30 sec, than allow it to settle. Save MIBK layer and discard aqueous layer.

6.3.10 Potassium Permanganate Solution, 5 percent (w/v), aqueous.

6.4 Procedure

6.4.1 Instrument Operation

6.4.1.1 It is difficult to formulate instructions as applicable to every instrument, because of differences between makes and models of atomic absorptions spectrometers. See manufacturer's operating manual.

6.4.1.2 Install a hollow-cathode lamp for silver in the instrument and set the wavelength at 328.1 nm. Set the slit width as suggested by manufacturer for silver being measured. Turn on the instrument and apply current to hollow cathode lamp for 10 min to 20 min to stabilize the energy source. After the adjustment of final position of the burner, aspirate the water-saturated MIBK solution into the flame and then gradually reduce the flow of the fuel until the flame is similar to the pre-aspiration of the solvent.

6.4.2 Standardization

A minimum of three concentrations of standard silver solutions (see 5.3.7) are required to be selected to bracket the expected silver concentration and to be, in the optimum range of concentration of the instrument, after extraction. The pH of 100 ml of a silver free water blank and 100 ml of standard is adjusted to a pH of 2 to 5 by addition of 1 N Nitric acid or 1 N sodium hydroxide, as required.

NOTE — For Silver extraction the most optimal pH range is 2.3 ± 0.2 .

Each type of standard solution and blank needs to be transferred into 200 ml of volumetric flasks, followed by addition of 1 ml of ammonium pyrrolidine dithiocarbamate solution to each of the flasks and shaken well to mix. Now add 10 ml of MIBK to each of the flasks, followed by vigorous shaking, for about 30 sec. (The maximum volume ratio of sample to MIBK is 40). Let the content of every flask to settle and separate into organic and aqueous layers, then carefully add water down the side of each flask to bring the organic layer to the neck so that it is accessible to the aspirating tube.

Set zero on the instrument at water-saturated MIBK blank. Now aspirate the organic extract into the flame directly and record the absorbance.

Prepare a calibration curve of absorbance vs concentrations before extraction on a linear graph paper.

6.4.3 Sample Analysis

Prepare the sample in a similar way, as the standards. Aspirate water saturated MIBK to rinse the atomizer. Now aspirate the organic extract as treated above into the flame directly and record the absorbance.

During the extraction, if any emulsion is formed at the interface of water-MIBK, add anhydrous sodium sulphate in order to obtain the homogeneous organic phase. In that case, add sodium sulphate needs to be added into all the blanks and standards. The silver need to be determined immediately after the extraction process in order to avoid the problems related to the instability of extracted complexes.

7 ELECTROTHERMAL ATOMIC ABSORPTION SPECTROMETRIC METHOD

7.1 General

This method is recommended for the determination of micro quantities of like silver. This method is applicable in the range from 1 $\mu\text{g/l}$ to 25 $\mu\text{g/l}$ of silver. However, the concentration range will vary with the sensitivity of the instrument used.

7.2 Apparatus

7.2.1 Atomic Absorption Spectrophotometer, the instrument should have background correction capability.

7.2.2 Graphite Furnace

Make use of an electrically heated device with some electronic control circuitry that is designed to carry

a graphite tube or cup with a heating program which provides sufficient thermal energy in order to atomize silver. For fresh waters with low dissolved solids content furnace heat controllers having three heating steps are enough. Furnace controller having upto seven heating steps programmed individually are used for brines, salt waters and other complex matrices. Adjust the furnace inside the sample compartment of the spectrometer instead of the assembly of the conventional burner. Make use of argon as a purge gas in order to minimize the oxidation of the furnace tube and to prevent the metal oxides formation. Also, use platform bearing graphite tubes in order to minimize the interferences and to improve the sensitivity.

7.2.3 Readout

7.2.4 Membrane Filter Apparatus, all glass filtering device and membrane filters having a pore diameter of 0.45 μm or smaller.

7.2.5 Cooling Water Supply, tap water at 1 l/min to 4 l/min to cool and use a recirculating cooling device.

7.2.6 Sample Dispensers, pipets of microliter capacity (5 μl to 100 μl) or an automatic sampling device which is designed for the particular instrument.

7.2.7 Source Lamps, a hollow - cathode lamp or an electrode discharge lamp.

7.2.8 Vent, place a vent about 15 cm to 30 cm above the burner to remove fumes and vapors from flame.

7.3 Reagents

7.3.1 Hydrochloric Acid, 1+1 and concentrated.

7.3.2 Nitric Acid, 1+1 and concentrated.

7.3.3 Silver Free Water, see **6.3.4**.

7.3.4 Matrix Modifier Stock Solution

7.3.4.1 Palladium nitrate, 4 000 mg Pd/l

Dissolve 9.34 g of palladium nitrate monohydrate in water and dilute it to 1 000 ml.

7.3.4.2 Citric acid, 4 percent

Dissolve about 40 g of citric acid in water and dilute it to 1 000 ml.

7.3.4.3 Magnesium nitrate, 10 000 mg Mg/l

Dissolve about 10.5 g of magnesium nitrate hexahydrate in water and dilute it to 100 ml.

NOTE — All the modifier solutions (see **7.3.4.1**, **7.3.4.2**, **7.3.4.3**) and can be prepared with the volumetric combinations of water and above solutions. In order to prepare the other matrix modifiers instructions by the manufacturer shall be followed.

7.3.5 Chelating Resin, 100 to 200 mesh, purified by heating at 60 °C in 10 N sodium hydroxide for 24 h. Cool the resin and rinse 10 times each with alternating portions of 1 N hydrochloric acid, silver free water, 1 N sodium hydroxide, and silver free water.

7.3.6 Stock Metal Solutions, see **5.3.7**.

7.3.8 Silver Free Sea Water (or Brine)

Fill a 1.4 cm inner diameter (ID) x 20 cm boronsilicate glass column with long structure to within 2 cm of the top having purified chelating resin. Now elute it with successive 50 ml portions of 1 N hydrochloric acid, 1 N sodium hydroxide, silver free water and silver free water at a rate of 5 ml/min just before its use. Pass the brine or salt water through the column at a rate of 5 ml/min so as to extract the trace metals present. Discard the initial 10 bed volumes (300 ml) of eluate.

7.4 Procedures

7.4.1 Sample Pretreatment

Pretreat all the samples before analysis. Wash all the volumetric glasswares with water and 1 + 1 Nitric acid. In order to avoid the contamination of the sample, perform the digestion procedures in a clean and dust free lab area in. For the silver determination no further pretreatment is required except addition of matrix modifier when necessary.

7.4.2 Operation of the Instrument

Align and mount the furnace device as per the instructions given in the manual provided by the manufacturer. Switch on the instrument and the data collection unit. After this, select an appropriate light source and then adjust it to the electrical setting as recommended. Now, select an appropriate wavelength of 328.1 nm and then select all the conditions as per the instructions given by the manufacturer. When the determination of elements is done at short wavelengths or when the samples have high level of soluble solids, background correction is highly required.

Select an appropriate inert or sheath gas flow. In few cases, it is desirable that the flow of gas is interrupted during the process of atomization. Such type of interruption leads to high level of sensitivity by increasing the residence time of the atomic vapor

in the optical path. Background absorption can also be increased by gas interruption and interference effects can be intensified, but these problems are usually eliminated by modern background correction methods. Make consideration of the advantages and disadvantages of the option for every matrix when the analytical conditions are optimized.

In order to optimize the conditions of the graphite furnace, precisely adjust the temperature settings of the furnace so as to maximize the sensitivity and precision and to minimize the interferences. Follow the instructions by the manufacturer.

Use the temperature for drying slightly above the solvent boiling point and provide sufficient time and temperature for the complete evaporation without spattering or boiling.

Select the temperature for atomization by the determination of the lowest temperature thereby providing the maximum sensitivity without eroding the precision significantly. Optimize the system by a series of successive analysis at various temperatures of atomization using a standard solution having absorbance between 0.2 to 0.5.

The temperature of charring must be high enough so as to maximize the volatilization due to interfering matrix components yet too low to volatize the elements under interest. With the atomization and drying temperatures set to the optimum values, perform the analysis of the standard solution at a series of charring temperatures in increments increasing between 50 °C to 100 °C. When the optimal point of charring temperature is exceeded, there will be a drop in sensitivity significantly. Draw a plot of charring temperature versus the absorbance of the sample: the optimal charring temperature is the highest without any reduced sensitivity. Verify the optimization with any kind of major changes in the sample matrix.

7.4.3 Calibration of the Instrument

Prepare the standard solutions by diluting the silver stock solution (5.3.7) for calibration of the instrument. After every two weeks, prepare the standard solutions or even more frequently as required if the response indicates the degradation of the standard. A decrease in signal by more than 10 percent requires investigation and indicates the need for the preparation of fresh standards.

Prepare a blank and minimum of three calibration standards in the appropriate range of 1 µg/l to 25 µg/l concentration for correlating the concentration of the silver and response of the

instrument. As closely as possible, match the matrix standard solutions to those of the samples. For brines or seawaters, make use of metal free matrix (7.3.8) as the standard solution is diluents. In addition to this, if needed, add the similar concentration of the matrix modifier to the standard solutions.

Inject an appropriate portion of each standard solution, in the order of their increasing concentration. Now analyze each of the standard solution in duplicate so as to verify the precision of the matrix.

On a linear graph paper, construct analytical curve by making a plot of the average peak absorbance or peak areas of the standard solution versus their concentration. Alternatively, also use the electronic instrument calibration in case the instrument is capable.

7.4.4 Sample Analysis

Carry out the analysis of all the samples except for those which are free of matrix interferences (based on recoveries of 85 percent to 115 percent for the concentration which is known) using standard addition methods. Carry out the analysis of all the samples at least in duplicate or till the results are reproducible. A variation of upto 10 percent is considered acceptable.

7.4.4.1 Direct determination

Inject a portion of pretreated measured sample in the graphite furnace. Take the same volume which was taken for the preparation of calibration curve. Add modifier immediately to the sample using automatic sampler and micropipette. Co-addition of it is also acceptable. For all samples and standards, use the similar volume and concentration of the modifier. Dry, char, and atomize as per the preset program. Repeat till the reproducible results are obtained.

Compare the peak area or the average absorbance value to the calibration curve to determine silver concentration. Directly read the results in case the instrument is equipped with capability. If the concentration or the absorbance or the sample peak area of the sample is more than the concentration (absorbance) or peak area of the most concentrated standard solution, dilution of sample is recommended and results are to be reanalyzed. In case very high dilutions are needed, another method like flame AA and ICP can be more suitable. During the final calculation, high dilution can magnify the small errors. Keep the modifier concentration (if present in the solutions) or acid background constant. Dilute the sample in a blank solution of matrix modifiers and acid.

7.4.4.1.1 *Calculation*

7.4.4.2 *Method of standard addition*

The standard addition method is valid only if it falls in the calibration curve linear portion. Once the sensitivity of the instrument has been optimized for the element to be determined and linear range has been established, carry out the analysis of the sample.

Into the furnace device, inject a measured value of sample. Dry, char or ash, atomize the samples as per the program that has been preset. Repeat the method till the reproducible results are obtained. Now, record the response of the instrument in concentration or absorbance as appropriate. Then add the known concentration of element to a separate sample portion so that the sample volume should not change significantly. Repeat the analysis.

Add a known concentration (twice of what is used in the first addition), to a separate portion of the sample. Mix it well and after that again carryout the determination.

NOTE — The steps followed should lead to the results in the linear range of the method.

Plot the average absorbance or response of the instrument for the sample and the additions on the vertical axis against concentration of the element

added on the horizontal one on a linear graph paper. Use zero as the concentration of the sample. Now, draw a straight line which connect the three points and then extrapolate it to the zero absorbance. The intercept on the horizontal axis is the negative of the concentration of the element in the sample. The concentration axis on the left of the origin should be the mirror image of the axis present on the right.

It is acceptable in case some instruments perform and calculate the method of standard additions.

7.4.4.2.1 *Calculations*

$$\text{Concentration of silver, } \mu\text{g/l} = C \times F \quad \dots(2)$$

where

C = concentration of metal as read directly from method of additions, in $\mu\text{g/l}$; and

F = dilution factor

8 INDUCTIVELY COUPLED PLASMA SPECTROSCOPY

Silver can also be determined by inductively coupled plasma optical emission spectroscopy with reference to procedure given in IS 3025 (Part 2). Likewise, inductively coupled plasma mass spectroscopy with reference to procedure given in IS 3025 (Part 65) can also be used for the determination of silver in water and wastewater.

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

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Amendments Issued Since Publication

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